Experimental Systems Overview

Many of the projects outlined in this section are also described in more detail in the individual beamline sections of this compendium. The intention here is to give an overview of most of the projects the Experimental Systems Group is working on and to highlight areas of special interest.

BEAMLINE 1.4 is designed for infrared microscopy and spectroscopy. Recently, light was extracted from the 40-mrad-aperture front end and successfully steered into the endstation interferometer and microscope (Fig. 1). Initial work concentrated on reducing vibration-induced intensity noise to an acceptable level, and with a combination of mounting the premirror chamber directly to the lower ALS floor, decoupling the M1/M2 mirror system from the shield wall, and minimizing the rf water pump vibration, the noise has been reduced to an acceptable level. Work is ongoing to reduce noise levels further, by techniques such as remounting the mirror switchyard away from the shield wall, but noise levels now are dominated by high-frequency electron-beam-induced motion. This phenomenon is under active study. The microscope is working well and is in routine use. Two other branchlines are under construction; the first is for surface science using the technique of surface infrared reflection spectroscopy, and it uses a Bruker interferometer. The second is for visible/UV spectroscopy of wide-bandgap materials, and it uses a small normal-incidence monochromator to provide a high-intensity beam. Both of these branchlines and endstations will be completed in the next few months.

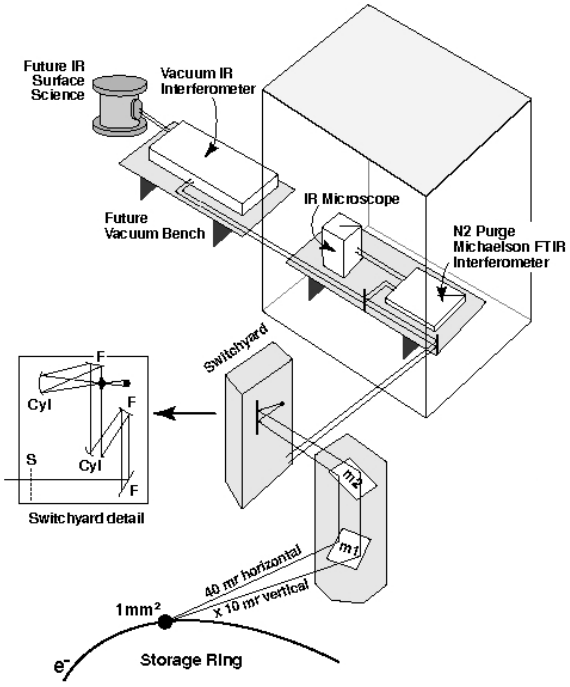

Figure 1. Schematic diagram of Beamline 1.4.

BEAMLINE 3.3.1 is designed for deep-etch lithography (LIGA). An aperture of 7 mrad is extracted through the shield wall and through a thick beryllium filter and window into a nitrogen-flowed end chamber at 16.7 m. The 100-mm-wide beam is used to illuminate a mask and photoresist-coated wafer mounted on a water-cooled carrier. The carrier is itself mounted on a stage that can oscillate up and down, so that the wafer is uniformly illuminated, and can rotate between exposure stations. The whole assembly is mounted in a steel radiation enclosure. The system was recently recommissioned and is now in routine use by a Sandia/Jet Propulsion Laboratory/Berkeley Lab Participating Research Team.

BEAMLINE 4.0.1 is designed for high-resolution soft x-ray spectroscopy of magnetic systems. The source is an elliptically polarizing undulator (EPU) that is in one-half of a chicaned straight section. The other half of the straight will eventually be occupied by a second EPU that will illuminate a second independent beamline, 4.0.2. The light passing down 4.0.1 is first deflected in the horizontal direction by a toroidal mirror, giving a vertical focus on the entrance slits of the monochromator and a horizontal focus in the user end chamber. The light is monochromatized by

an entrance-slitted Peterson-style plane-grating monochromator. This has a vertically reflecting variable-angle plane premirror and plane grating and a horizontally deflecting cylindrical mirror to focus in the vertical direction on the exit slits. The light is then refocused in the vertical direction after the slits by a variable-radius cylindrical mirror. The main challenge in this system is to deal with the extreme power density at the monochromator premirror while maintaining an extremely small slope-error budget. This has necessitated using an internally cooled silicon premirror with aggressive mini-channel multipass cooling. Due to the wide energy range (20–1800 eV) of the monochromator, the variable-angle premirror has to be long (600 mm optical length), and this compounds an already difficult design. The monochromator is being manufactured by Oxford Instruments and is due to be commissioned with light in September 1998. The undulator itself has adjustable phasing so that horizontal, vertical, right- or left-hand circularly polarized light can be selected by the user.

BEAMLINE 5.0.1, a side branch off the 37-pole, 2.1-Tesla wiggler beamline designed for protein crystallography, is under construction. To provide vertical focusing, there will be an upwards-deflecting, 1-m-long, superpolished, Ni-plated Glidcop mirror inside the shield wall, and this will be dynamically bent as well as internally cooled. Light passes through the existing Beamline 5.0.2 monochromator and is deflected horizontally by an asymmetric-cut curved-crystal monochromator. This also will be internally water cooled and will use five separate crystals of differing asymmetric cuts to limit the focal-plane motion to small values. These crystals will be mounted on a linear vertical stage to allow rapid interchange. The monochromator will cover the range from approximately 8.5 keV to 14 keV.

BEAMLINE 5.0.2 was commissioned in November 1997 and is now in routine use. It has the same premirror configuration as 5.0.1, except that in this case the beam is deflected downward. Upstream of the premirrors, a carbon filter system is used to remove unwanted low-energy light to reduce the power load on the components that follow. The beamline uses an Oxford Instruments double-crystal constant-exit-height monochromator, with mini-fin water-cooled Si[111] crystals designed in-house, and is used up to around 14 keV. The endstation is equipped with a Newport Kappa diffractometer and a 2×2-matrix CCD x-ray detector from Area Detector Systems Corporation (Fig. 2). Due to vendor problems, the beamline is currently equipped with temporary mirrors that are shorter than those in the final configuration and do not have the small slope error that is needed to produce a small vertical focus. The beam size at the sample is around 0.5 mm FWHM in both directions; this is correct in the horizontal direction but is too big by about a factor of 2 in the vertical direction. The beamline accepts only 40% of the vertical aperture of the full-length mirrors, and excessive surface roughness reduces the combined reflectivity of the mirrors by a factor of 2. The overall flux density is therefore approximately one order of magnitude what it will be with the final mirror system. Even so, the flux measured in the 0.5-mm focus is approximately 1×10^{12} photons/s at 12.4 keV, and this gives typical data collection times of around 2 hours for 0.3-mm crystals. In addition, microcrystals as small as $30 \times 40 \times 5$ μm have been successfully measured. A further development is that a prototype vertical microfocus mirror has been tested and has successfully demagnified the vertical beam size from 0.5 mm to 0.035 mm FWHM, giving a measured flux density increase of a factor of 7. A dedicated version of this mirror system is currently under construction.

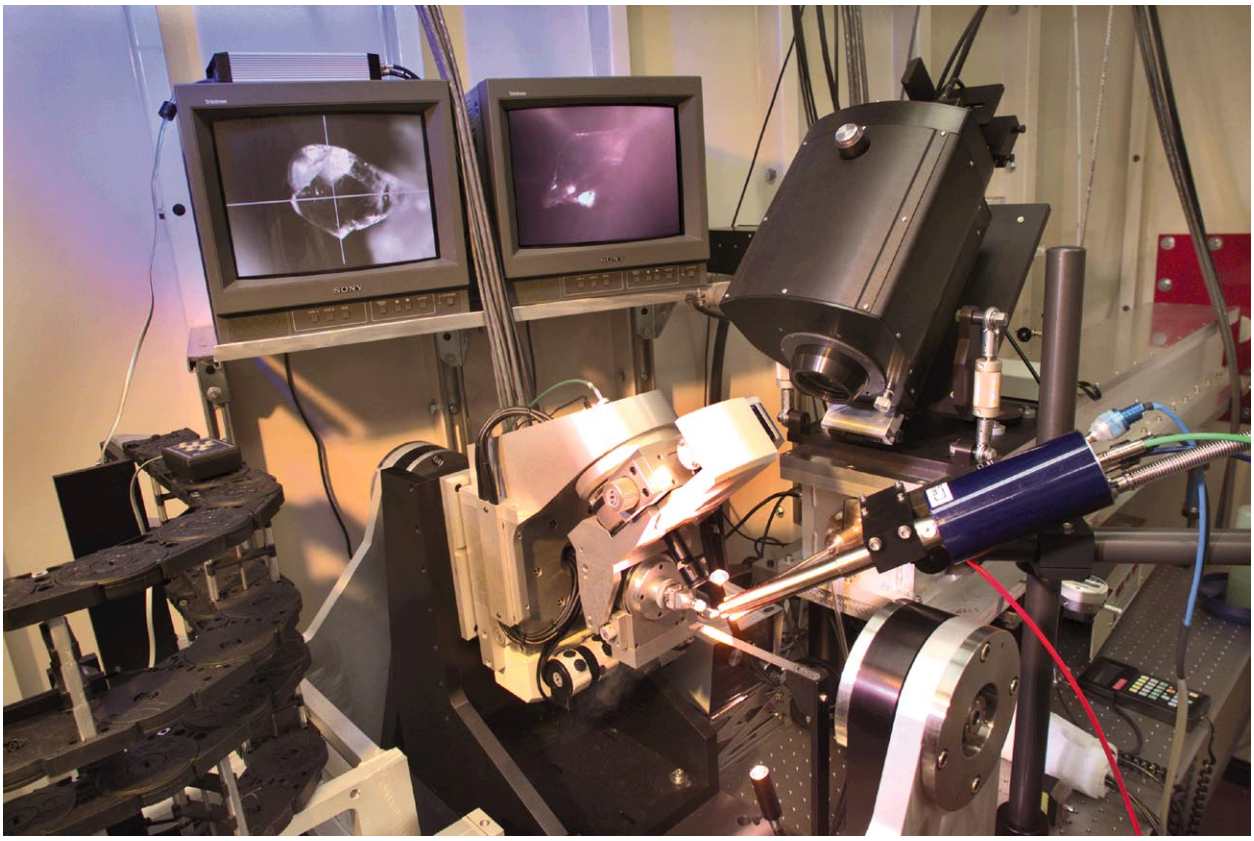

Figure 2. Photograph taken inside the Beamline 5.0.2 hutch.

BEAMLINE 7.0 is in use for Participating Research Team and independent investigator research 24 hours of every operational day. It has also been the site for scanning zone-plate microscope development. Two scanning zone-plate microscopes have been developed for x-ray spectroscopic analysis of materials. A focused x-ray spot is rastered over the sample to make an image, then held on a feature of interest for spectral measurements. The count rates in these microscopes are about ten times higher than previously available. We are currently using zone-plate lenses with a central stop and 80-nm outer zone width, and a corresponding diffraction limit to the spatial resolution of about 100 nm. An order-sorting aperture (OSA) is held in front of the sample and precisely positioned on the optical axis (+/-2 μm) to allow only the first-order diffracted focus to reach the sample. The measured FWHM of the x-ray spot is 150 nm.The Scanning Transmission X-ray Microscope (STXM) provides imaging NEXAFS analysis of samples in transmission at atmospheric pressure. The transmission geometry is the most efficient use of photons for an absorption spectrum, well suited to radiation-sensitive organic samples. Circularly polarized photons have been generated and used for imaging domains at the L edge in Fe, Ni, and Co magnetic films. The Scanning Photo-Electron Microscope (SPEM) provides imaging XPS and NEXAFS analysis of sample surfaces in a UHV environment. Here the sample is stationary during imaging and the zone plate is rastered in the illumination field to carry the focused spot across the sample surface. NEXAFS capability is included by means of a UHV flexure to carry the zone plate 0.5 mm longitudinally to retain the focus condition as the photon energy changes. Different photon energies require different zone-plate/OSA combinations with different built-in focal lengths. So far, we have operated the microscope with three zone-plate/OSAs aligned in this way. SPEM allows us to perform quantitative XPS measurements of atomic concentration and core-level chemical shifts over regions of the sample surface as small as the spatial resolution of the zone-plate lens. The

zone-plate array can be lowered out of the beam, and the sample surface can be observed with a magnifying video system, allowing visible fiducial marks on the sample to be used to position the region of interest within the 100- × 100-μm range of the scan stage. XPS spectra are measured with typical photo-peak count-rates of 70,000 counts/s (Au 4f at 420eV photon energy).

BRANCHLINE 7.3.1.1 uses Beamline 7.3.1's single spherical grating, which provides monochromatic light to two endstations, photoemission electron microscopy (PEEM) on Branchline 7.3.1.1 and micro x-ray photoelectron spectroscopy (μXPS) on Branchline 7.3.1.2. The PEEM beamline accepts 2 mrad of horizontal aperture and uses a 1-m-long horizontally deflecting elliptical mirror to demagnify the source at 10:1 onto the sample (Fig. 3). The grating images at a monochromatic demagnification of 1:1, so with a source size of 250 μm (horizontal) × 22 μm (vertical), we should see a monochromatic image of 25 × 22 μm in the endstation.

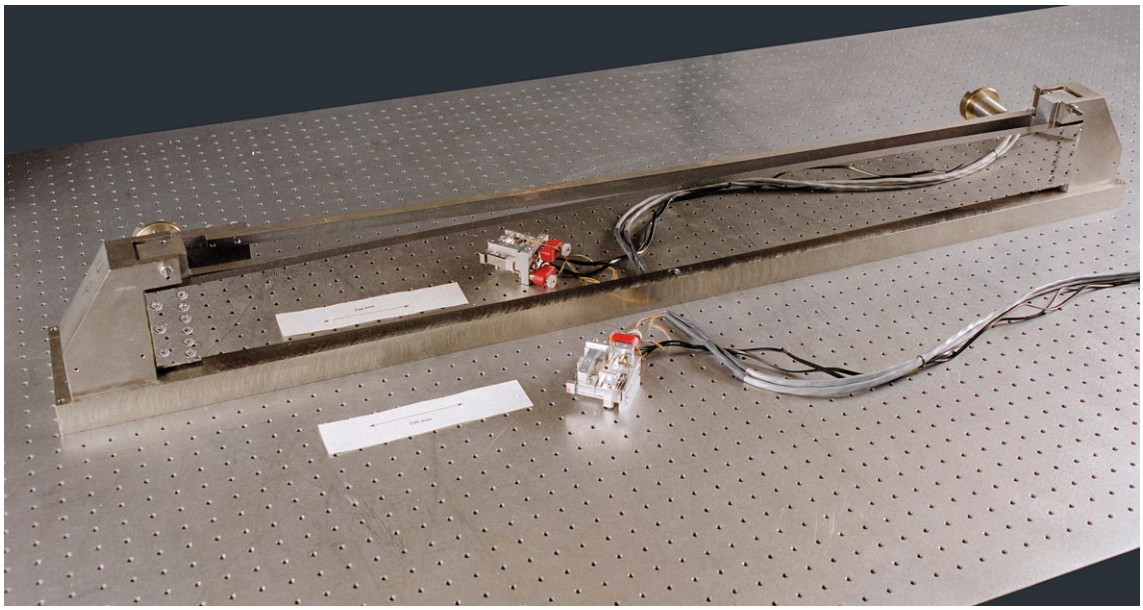

Figure 3. Photograph of the 1-m-long PEEM mirror alongside a smaller Kirkpatrick-Baez mirror for the μXPS experiment.

The measured horizontal width is 30 μm FWHM, and this has to be compared to the 40-mm width of the beam 1.85 m upstream before focusing! This extreme focusing is only possible with elliptical surfaces, and a 1-m-long mirror with the required ellipticity is well beyond the state of the art for conventional optical grinding. We therefore chose to produce the mirror by controlled bending of an initially flat surface by the application of unequal couples to a nonuniform-width beam. This proved to be a challenging project, but as evidenced by the superb focus, we now have developed a very powerful and widely applicable mirror manufacturing method. The 7.3.1.1 PEEM beamline requires as high a flux density as possible in a 20- or 30-μm spot size to enable high-resolution XMCD microscopy to be conducted with reasonable imaging times. Under a cooperative research and development agreement (CRADA) with IBM Almaden, we have constructed a high-voltage PEEM for magnetic imaging using circularly polarized light (Fig. 4). The beamline and PEEM are now complete, and the first magnetic surface images have been recorded. The microscope should be capable of 20-nm resolution, and therefore getting sufficient flux into a 20-nm sample element to correspond to reasonable MCD imaging times is a major challenge. The beamline is performing near its design flux density, so the remaining challenge is to fully commission the microscope to achieve its design resolution target. The 30-kV PEEM

microscope design paid particular attention to thermal and mechanical stability and has several novel features, such as interchangeable back focal plane apertures, direct-conversion imaging, and a V-lens mounting system. The microscope also has a sophisticated sample transfer and preparation system.

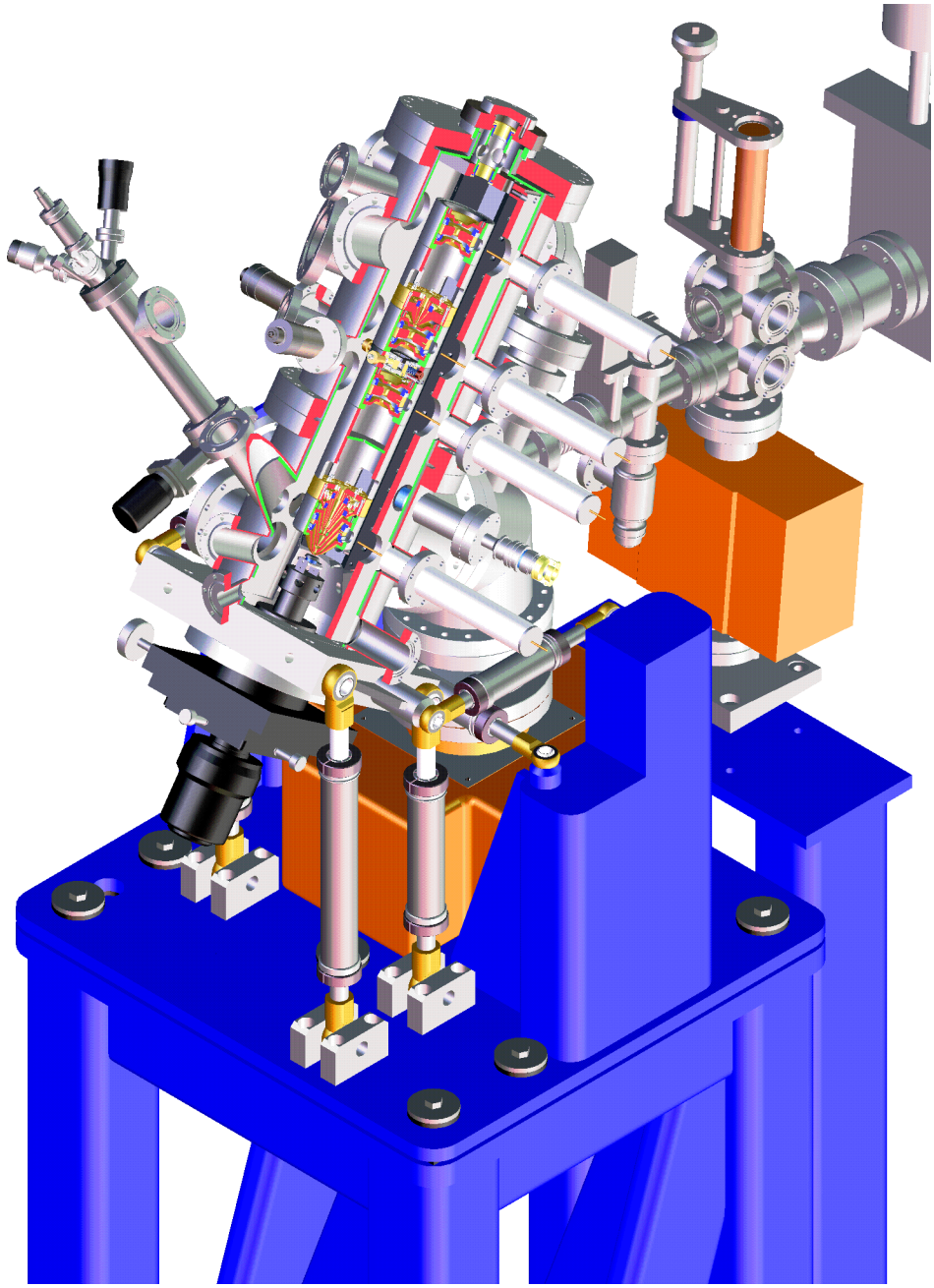

Figure 4. Three-dimensional engineering drawing of the PEEM microscope.

BRANCHLINE 7.3.1.2 is designed for x-ray photoelectron spectroscopy with a spatial resolution of 1 μm (μ-XPS), with tunable photon energies up to 1.3 keV. It uses a horizontal branch mirror just downstream of the Beamline 7.3.1 grating to deflect and focus 0.2 mrad of aperture to the monochromatic focus position of the grating. A pair of bilaterally adjustable slits defines a 20 (v)- × 40 (h)-μm object, and a pair of Kirkpatrick-Baez elliptically bent mirrors is

used to demagnify to a 1-μm focus. These mirrors are particularly challenging, as they have to be inside the main experimental end chamber to achieve the required 20 and 40:1 demagnifications, and they have to be integrated with the sample interchange system, electron analyzer, neutralizer, ion gun, and other surface science equipment. The system now works routinely at 1-μm spatial resolution and, as importantly, wafer samples can be introduced into the chamber and optically refiducialized to the same accuracy. Combined with laser interferometric recording of the sample puck location, we can drive the sample over its 50- × 50-mm size to micron absolute accuracy. This system was developed in collaboration with Intel and Applied Materials and is being applied to a wide range of semiconductor manufacturing issues.

BEAMLINE 7.3.3 is being developed for microfocus x-ray diffraction/spectroscopy and ultra-fast time-resolved x-ray diffraction/spectroscopy. The beamline consists of a grazing-incidence toroidal mirror that collects 3 × 0.2 mrad of bending magnet radiation and focuses it to a ~300- × 200-μm (FWHM) spot in the experimental x-ray hutch. The 0.7-m-long silicon mirror is shown in Figure 5 below. This mirror is bent to the correct major radius of about 3 km by means

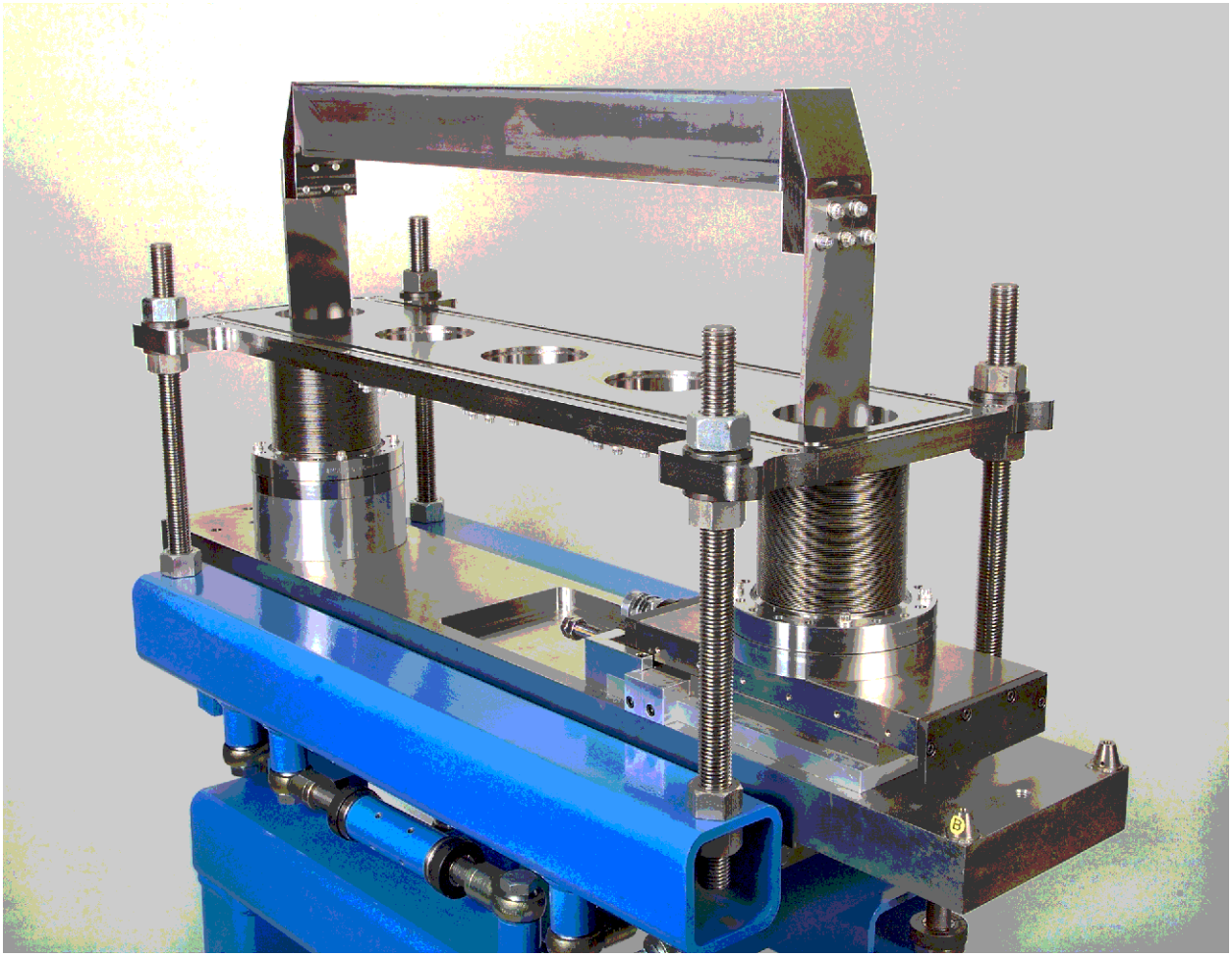

Figure 5. Photograph of the 0.7-m-long bendable mirror for Beamline 7.3.3.

of the flexural springs that support the ends of the mirror. These springs are driven together at the base support structure and provide the necessary force for the mirror bending. The operating angle is 5.4 mrad, which with the platinum coating, allows for useful flux up to 15 keV. There are two

experimental stations in the x-ray hutch. The first experiment station (Roger Falcone, Univ. of Calif., Berkeley) combines x rays with a femtosecond laser system and time-resolved detectors. X-ray diffraction and absorption are utilized as probes of laser-induced phase transitions. The second experiment station is under construction and is designed to carry out x-ray diffraction and x-ray absorption on micron-sized samples. In operation, the grazing-incidence toroidal mirror described above images the source onto some slits at the hutch entrance. These slits provide a source of adjustable size for some Kirkpatrick-Baez (K-B) mirrors that refocus this secondary source onto the sample with spot sizes in the micron range. As the secondary source is adjustable in size, flux can be traded for spot size on the sample. A new custom goniometer with a large-area x-ray CCD is to be installed as the endstation. The operational procedure for carrying out x-ray microdiffraction is to be the same as that established on the development beamline, 10.3.2.

BEAMLINE 10.0.1 is designed for high-resolution spectroscopy in the 20–350 eV range. The beamline will reuse many of the components, including the high-resolution spherical-grating monochromator, from beamline 9.0.1 (which was decommissioned in March, 1998) to provide the high spectral resolution ($E/\Delta E \geq 10{,}000$) and photon flux ($\geq 10^{12}$ photons/s) required by the users. A full-length 10-cm-period undulator built at the ALS will be installed during the spring 1998 shutdown to provide a photon source for the beamline. The horizontal focusing optics from beamline 9.0.1 will be replaced to produce small beam spot sizes (100–400 μm) for the two side branches of the beamline while providing a highly collimated beam with a width of approximately 1 mm for the central branch. Two projects funded by the DOE Scientific Facilities Initiative necessitated the construction of this new undulator beamline: (1) electron spectroscopy of highly correlated materials, and (2) high-resolution atomic, molecular, and optical spectroscopy. Endstations for both projects are under construction and will be permanently positioned at the end of Beamline 10.0.1.

BEAMLINE 10.3.2 is being used in collaboration with the Center for X-ray Optics (CXRO) and Earth Sciences Division (ESD) as the x-ray optics development beamline for the techniques of micro x-ray diffraction and micro x-ray absorption. The beamline arrangement is a 4-crystal monochromator that can pass both white and monochromatic light along the same axis, followed by a pair of K-B mirrors that have been used to image the bending magnet source to spot sizes down to 0.8 μm (FWHM) on the sample. The x-ray microprobe can be switched from white light to monochromatic light whilst illuminating the same micron-sized sample.

Figure 6 shows the Laue pattern from a single aluminum grain in an aluminum wire on a silicon semiconductor wafer recorded in 0.5 seconds. The dominant symmetric pattern is from the silicon substrate, but the asymmetric pattern indicated and indexed is that of the micron-sized aluminum grain of interest. The interest is one of determining the strain within the aluminum grain, as this will provide some insight into the electromigration problem that significantly affects chip reliability in the semiconductor industry. The strain can be measured if one accurately knows the d spacing of the various Laue spots. By switching to monochromatic light and determining the angle of the Laue spot, an accurate measure of the d-spacing can be obtained.

In the case of micro x-ray absorption, the technique is to carry out an elemental scan with either white or monochromatic light, followed by x-ray absorption spectroscopy on the area of interest. Figure 7 shows the micro x-ray absorption spectrum from a live fungus root contaminated with zinc. The elemental map indicates small regions (<5 μm) of high zinc concentration. The absorption spectra show the zinc K edge from the fungus and that from a zinc oxalate standard.

They appear to be very similar, which suggests that zinc oxalate is important in the mechanism of contaminant uptake, retention, or conversion in this fungus metabolism.

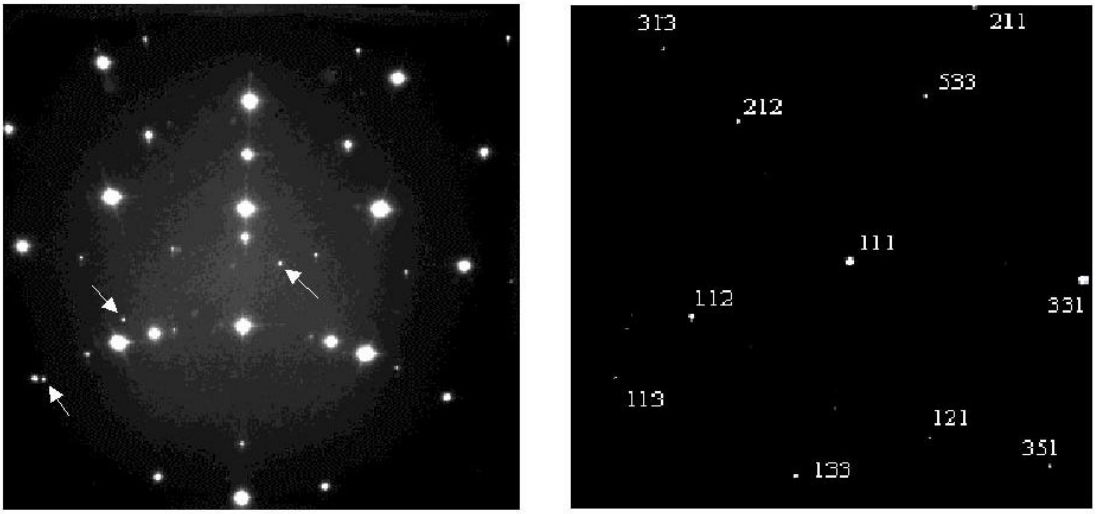

Figure 6. Laue diffraction pattern of a single aluminum grain on a silicon substrate (left, sample provided by Intel). Arrows indicate three of the aluminum diffraction spots. Digitally subtracting the dominant silicon spots shows the entire aluminum Laue pattern that can then be indexed (right).

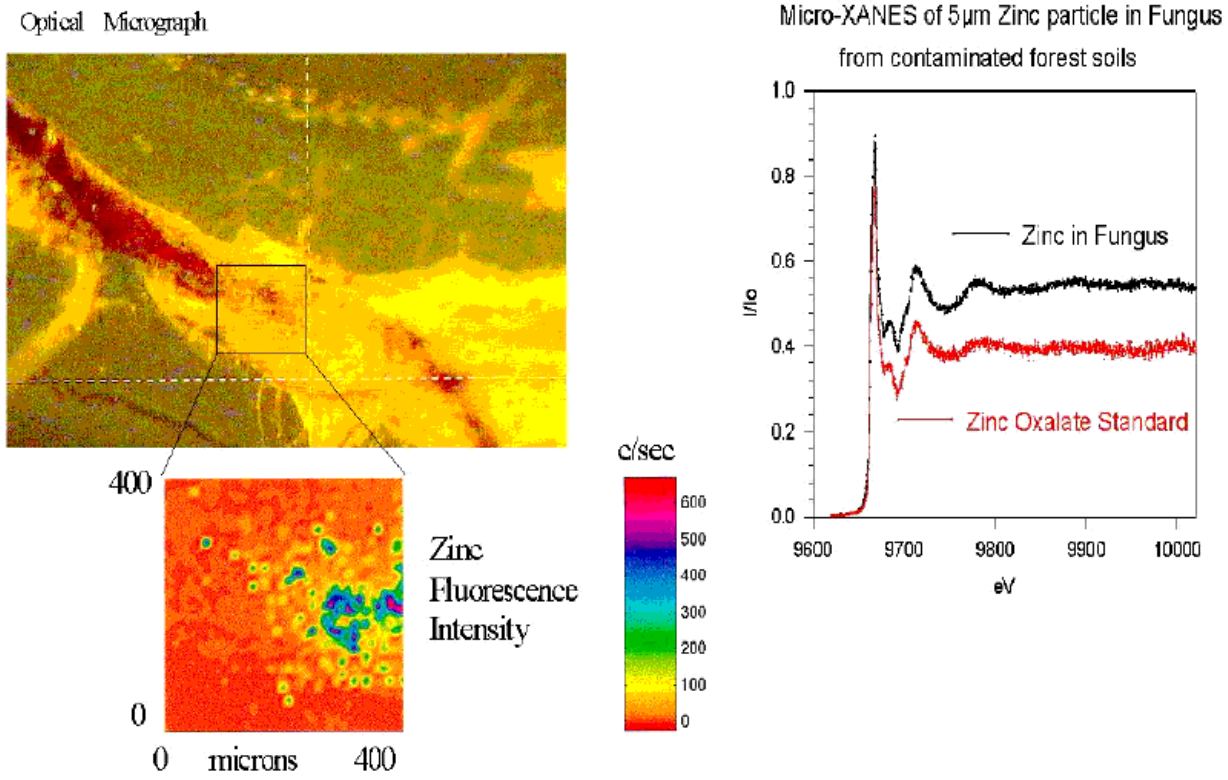

Figure 7**.** Optical micrograph of fungus in its natural state (top left) with Zinc elemental fluorescence map (lower left). Also included are the near-edge zinc XAS spectra of the high-concentration zinc particles and of a zinc oxalate standard.